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APPLICATIONS OF FOURIER TRANSFORM INFRARED PHOTOACOUSTIC SPECTROSCOPY TO SOLID PROPELLANT CHARACTERIZATION

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AUGUST 1991

U.S. ARMY LABORATORY COMMAND

BALLISTIC RESEARCH LABORATORY
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Fourier transform infrared photoacoustic spectroscopy (FT-IR-PAS) has been used to examine surfaces of composite solid propellants. Depletion of crystalline energetic material from extruded surfaces and of plasticizer from aged surfaces has been documented. In addition, a method has been developed to determine the mass per unit area of graphite on coated propellant surfaces.

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1. INTRODUCTION

Observations by members of the propellant community have generated interest in the examination of solid propellant surface composition (Rocchio, private communication 1988). Such observations include occasional lot-to-lot differences in performance tests (e.g., flamespreading) for propellants with identical formulations or between aged and unaged propellant. Along with the interest in surface composition comes the need for experimental techniques capable of detecting surface variations resulting from such phenomena as extrusion "wiping" effects, plasticizer evaporation and diffusion, and nonhomogeneous application of the graphite commonly used to coat propellant grains. Although all of these phenomena are potentially important, none has yet been characterized or correlated with ballistic behavior, primarily because of the absence of suitable experimental surface characterization techniques. Conventional chemical analysis techniques require the accurate removal of micrometer-thick layers from the sample surfaces. Removal of such thin layers from the surface of brittle propellants is not readily accomplished without concomitant surface modification.

In this investigation, the above-mentioned phenomena were examined by Fourier transform infrared (FT-IR) photoacoustic spectroscopy (PAS), a nondestructive technique for near-surface analysis (McClelland 1983; Grahm, Grim, and Fateley 1985). There are two main advantages of FT-IR-PAS; first, depth profiling can be performed by obtaining spectra at varying interferometer mirror velocities; and second, sample preparation is usually not required (Michaelian 1989; Urban and Koenig 1986; Yang, Bresee, and Fateley 1987). The ability to analyze samples with virtually no preparation proved to be extremely useful for the analysis of these composite propellant samples. The only requirement for this study was that samples fit in the sample holder. Because our interest was the composition at extruded surfaces, it was important to perform the analysis with minimal sampling handling. With FT-IR-PAS, sample handling is minimized, and complications that are introduced by analysis with more traditional methods are avoided. Results demonstrating the applicability of FT-IR-PAS to characterization of solid propellants are described in this report.

2. EXPERIMENTAL

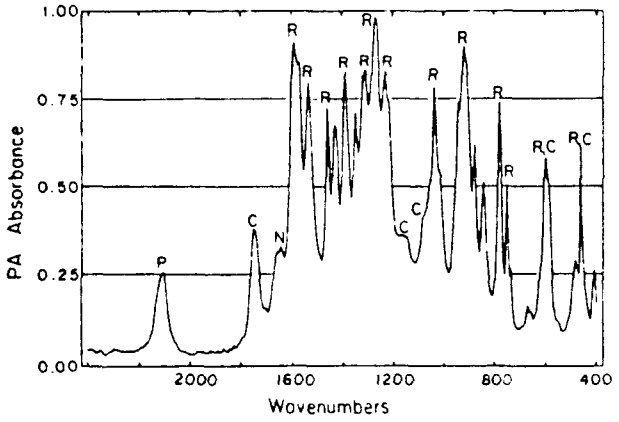
Spectra were obtained on a Mattson Polaris FT-IR spectrometer using software provided by the manufacturer. Detection of the photoacoustic signal was achieved with a helium-purged MTEC Model 100 photoacoustic cell. Each spectrum was the average of 32 scans with a resolution of 8 cm^{-1}. Spectra were obtained with a moving mirror velocity of 0.316 cm/s and were ratioed against a carbon black (Norit-A) background.

The composite propellant samples used in this investigation were cylindrical in shape with a length and diameter of 1.4 and 1.1 cm, respectively, and are commonly referred to as "grains." The formulation was composed of crystalline energetic material (cyclo-1,3,5-trimethylene-2,4,6-trinitramine, RDX), non-energetic binder (cellulose acetate butyrate [CAB]), energetic binder (nitrocellulose [NC]), antioxidant (ethyl centralite [EC]), and either of two plasticizers. One is an azide plasticizer, hereafter referred to as "P," whose complete structure is proprietary information. The other is a mixture of two dinitro plasticizers, hereafter referred to as "A/B," whose structures are also proprietary information. Grain exteriors commonly have a thin graphite coating which provides lubrication when packing into artillery shells and protects against static electrical charges that might otherwise result in unintentional ignition.

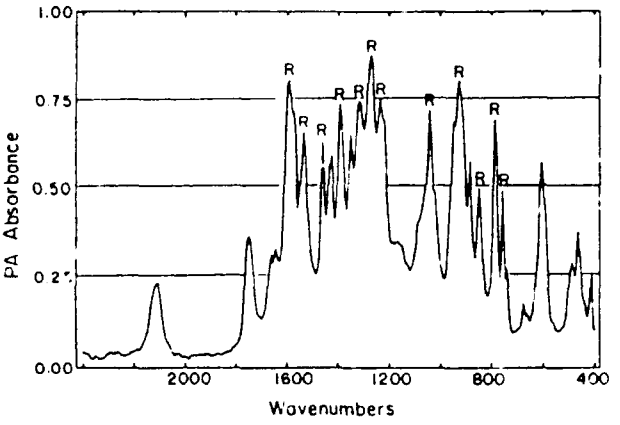
3. RESULTS AND DISCUSSION

3.1 Depletion of RDX at Extruded Surfaces. Photoacoustic spectra of a typical sample are given in Figure 1. Bands assigned to RDX (R), cellulose acetate butyrate (C), nitrocellulose (N), and plasticizer (P) are appropriately labeled in spectrum A. Spectra B, C, and D are labeled to draw attention to specific bands.

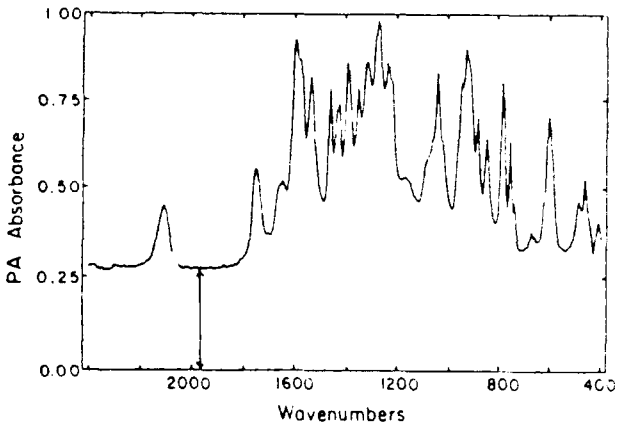
Comparison of spectra A and B indicates a reduced level of RDX at the extruded surface, as evidenced by the decreased relative intensity of RDX bands. This reduction is attributed to the wiping action of the extrusion process which covers RDX particles with polymeric binder at the propellant surface. This finding supports conclusions based on scanning electron microscopy of extruded propellant surfaces (Lieb, private communication).



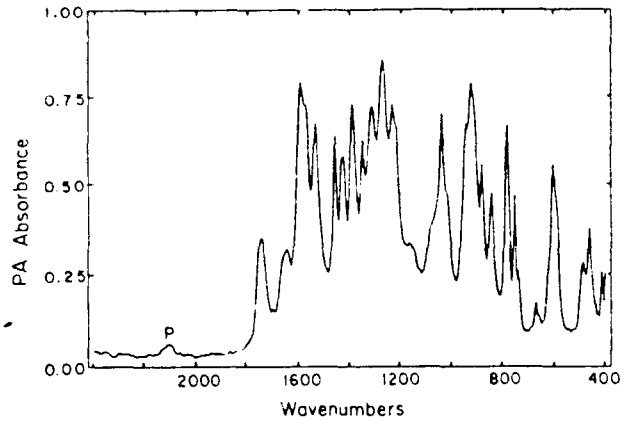
(A) Bulk Surface (cleaved)



(B) Side Surface Without Graphite Coating



(C) Side Surface With Graphite Coating



(D) Uncoated Side Surface After Accelerated Aging

LEGEND: R = RDX, C = Cellulose Acetate Butyrate, N = Nitrocellulose, P = Plasticizer

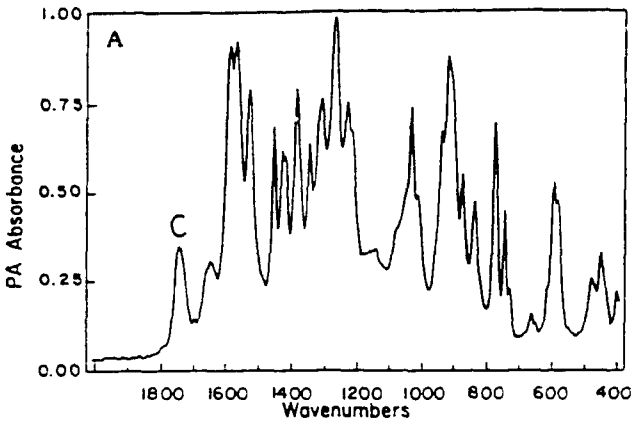
Figure 1. FT-IR-PA Spectra of Propellant Grains.

3.2 Plasticizer Loss From Aged Surfaces. Spectrum 1D illustrates the applicability of FT-IR-PAS to the examination of propellant aging. This spectrum was obtained from the extruded surface of a propellant grain that had been heated at 80° C for 26 hours. Loss of plasticizer "P" is demonstrated by the large decrease in absorbance of the plasticizer's azide band at 1,900 cm^{-1}. The same phenomenon has been observed for composite propellants formulated with several other plasticizers. Depletion of plasticizer has been observed at room temperature as well as at elevated temperatures. Spectral conditions are reproducible over long time periods, making both long term ambient and short term accelerated aging studies possible.

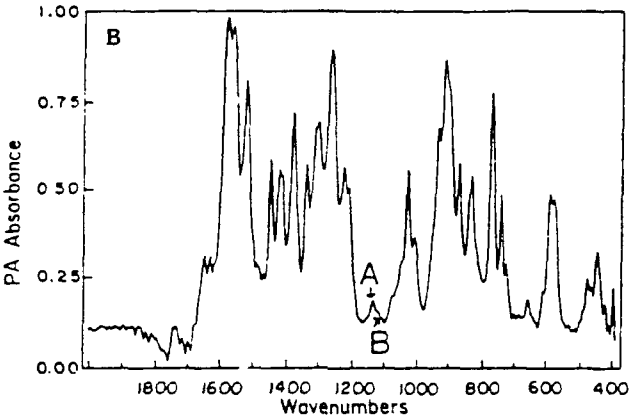
An example of depletion of "A/B" plasticizer is given in Figure 2. Although the infrared spectra of the two dinitro plasticizers are very similar they can be distinguished by bands appearing between 1,200 and 1,100 cm^{-1}. In propellant spectra, these bands are obscured by CAB bands but can be observed by performing digital subtraction (i.e., propellant spectrum minus CAB spectrum). The spectra in Figure 2 illustrate the loss of "A/B" plasticizer on heating.

3.3 Graphite Surface Coating Analysis. An interesting application of FT-IR-PA spectroscopy to the characterization of solid propellants is the analysis of their graphite coating. A method has been developed to determine the mass of graphite on a given surface area. Such information is useful in understanding lot-to-lot variations in propellant ignition and flamespreading. For cases where quantitative measurements are not necessary, relative graphite "thicknesses" can be readily obtained by simply comparing the baseline absorbance of FT-IR-PA. As illustrated in Figure 1C, the spectral baseline of coated propellant surfaces occurs at a higher photoacoustic absorbance value than that of uncoated surfaces (see Figure 1A).

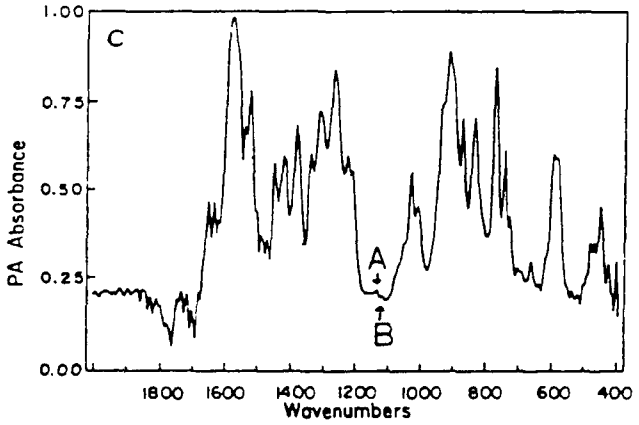
This observation is explained by considering that graphite, a black body absorber, gives a relatively flat spectrum when its single-beam spectrum is ratioed against the carbon black background spectrum. For coated grains, the FT-IR-PA spectrum includes both the flat graphite spectrum and the spectrum of the propellant layer below the graphite. It was found that the baseline "absorbance" value was proportional to the mass of graphite on the propellant surface. With this information, a method was developed to determine the graphite mass per unit area.



(A) Untreated Propellant Spectrum



(B) Untreated Propellant Spectrum Minus CAB Spectrum



(C) Heat-Treated Propellant Spectrum Minus CAB Spectrum

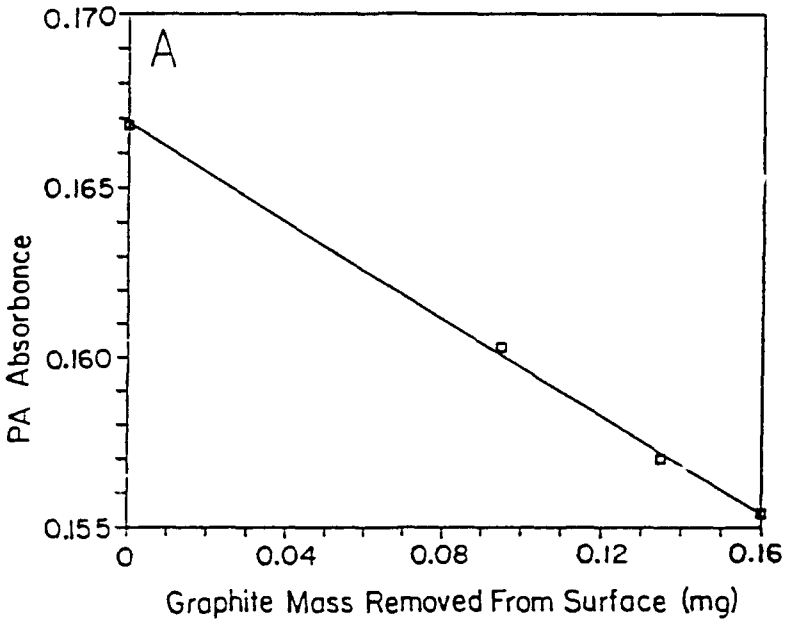
LEGEND: C = Cellulose Acetate Butyrate; A and B Are the Two Dinitro Plasticizers.

Figure 2. FT-IR-PA Spectra of Propellant Grains Plasticized With "A/B" Plasticizer.

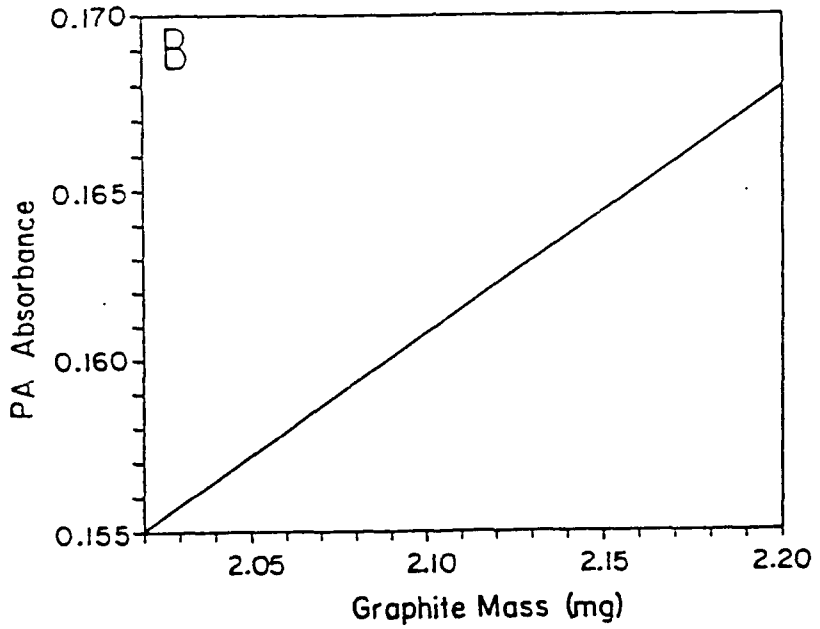
The method requires the construction of a calibration curve whose slope is the ratio of photoacoustic baseline "absorbance" to graphite mass and whose intercept is the baseline "absorbance" for an uncoated sample. Determination of the calibration curve slope is not straightforward, as standards for such a determination are not available. For this reason, the slope was determined indirectly by following the decrease in baseline "absorbance" as graphite was removed from a coated propellant surface. Graphite removal was achieved by gently wiping the coated surface against a piece of preweighed filter paper. Quantitation of transferred graphite was obtained from the difference in filter paper mass after transferral. Measurements were made to +/- 0.001 mg with a Perkin Elmer AD2Z Autobalance. The graphite removal procedure was repeated no less than four times on the same coated slab and discontinued before the underlying layer of propellant was exposed. Confirmation that only graphite was removed from the slab was obtained by examination of the FT-IR-PA spectrum of the filter paper after transferral.

Figure 3A gives typical results of the above procedure, i.e., the relationship between photoacoutic absorbance and graphite mass removed from the propellant surface. Only a small fraction of the graphite was removed from the sample surface to generate the data in Figure 3A. Removal of additional graphite would risk concomitant removal of propellant in the layer below. The slope of the calibration curve is then calculated by multiplying the slope of the line in Figure 3A by -1, which then gives the relationship between photoacoustic absorbance and increasing mass of graphite on the propellant surface. For completion of the calibration curve, a baseline absorbance measurement for uncoated propellant is made and taken as the Y intercept. The resultant calibration curve is given in Figure 3B. With this calibration curve and samples with known surface area, routine analysis of coated grains may be performed. Values obtained from the analysis have units of mass of graphite per unit area.

Using this method, propellant grains coated with a set of ten graphite samples, varying in particle size and crystallinity, were analyzed. Exposure of reproducible surface areas was ensured by placing a circular brass mask over the propellant sample. The mask was contoured to the cylindrical shape of the grain to minimize the cell volume and prevent loss of the acoustic signal. Slopes of calibration curves were determined from at least three samples with each type of graphite. Measurements of mass of graphite on a given surface area were obtained from at least six samples with the same type of graphite. Results of the analysis are given in in Table 1.



(A) PA Absorbance vs. Mass of Graphite Removed From Propellant Grain Surface



(B) PA Absorbance vs. Mass of Graphite Remaining on Propellant Grain Surface

Figure 3. Experimental Data and Calibration Curve for Graphite Coating Analysis.

Table 1. Summary of Graphite Glaze Analysis

Graphite Source and Number	Graphite Type (% ash)	Graphite Particle Size (μm)	PA Absorbance at 2,000 cm^{-1}	Calibration Curve Slope	Graphite Mass per Unit Area (mg/cm^2)
Dixon 200-10	natural (2)	2.5	19.1	9.8	5.2
Dixon 200-39	synthetic (0.5)	2.5	18.3	14.7	3.3
Dixon 200-19	amorphous (19.4)	2.5	16.2	9.9	4.2
Dixon 400-10	natural flake (2.9)	2.5	17.5	11.9	3.9
Asbury Mills 041-C	natural (3.2)	4.0	18.0	11.9	3.9
Asbury Mills M150	amorphous (17.2)	4.3	19.8	9.3	5.5
Asbury Mills M450	synthetic (0.5)	4.3	18.5	12.8	3.9
Dixon 041-B	natural (?)	7.5	16.0	12.2	3.3
Dixon 041-A	natural (?)	18.0	16.3	8.8	4.7
Dixon 155	natural flake (4.3)	18.0	17.1	13.4	3.3

Calculated values for mass of graphite per unit area fall within a small range of values (i.e., 3.3 to 5.5 mg/cm^2). Values for calibration curve slopes can be divided into at least two groups, i.e., those with slopes 8.8 to 9.9 and those with slopes of 11.9 to 14.7. Although the relationship between the calibration curve slope and graphite type has not yet been determined, it does make sense that the former group includes two samples with high ash content (Dixon 200-19 and Asbury Mills M150 have 19.4 and 14.7% ash, respectively). Ash is typically composed of silica, alumina, iron oxide, calcium oxide, potassium oxide, sodium oxide, sulfur trioxide, and sulfuric anhydride, none of which absorb radiation at 2,000 cm^{-1}. Removal of graphite with a high ash content will result in a smaller change in absorbtivity than will the removal of graphite with little or no ash. The two other samples in the 8.8 to 9.9 slope group, Dixon 200-10 and Dixon 041-A, have less than 5% ash. It is not clear why their calibration curve slopes are lower than other samples with low ash content.

3.4 Additional Applications. As part of a study of condensed phase processes during solid propellant combustion (Schroeder 1990), we are using FT-IR-PAS to examine samples that have been ignited and subsequently extinguished. For example, for RDX/CAB propellants, we observe that the extinguished surface is essentially a foamy layer of binder (cellulose acetate butyrate). The foam is believed to have formed by the flow of gaseous RDX decomposition products through molten binder. Substantially reduced levels of RDX and plasticizer were observed for this layer. Material below the foamy layer was found to have a composition similar to that of unburned propellant.

In another study, FT-IR-PAS was used to analyze the bulk composition, rather than the surface composition, of two explosive samples composed of RDX, stearic acid, magnesium oxide, and graphite. Although the formulations were believed to be identical, ballistic properties of the two samples varied greatly. Photoacoustic spectra indicated not only differences in the stearic acid level but also the presence of residual cyclohexanone solvent in one of the samples. As is typical for most investigations of solid composite propellants by FT-IR-PAS, the analysis provided qualitative and semi-quantitative results relatively quickly and required no sample preparation.

4. CONCLUSIONS

The applications of FT-IR-PAS described above illustrate how well suited the technique is for characterization of composite propellant surfaces. The ability to detect variations in surface composition caused by plasticizer evaporation, extruder "wiping" effects, and nonhomogeneous application of graphite, provides a useful tool for understanding surface-related inconsistencies in ballistic behavior. Because so little sample preparation is required, FT-IR-PAS has the potential to be incorporated into both quality control/quality assurance monitoring and developmental studies of solid propellants.

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